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## Development of Low Modulus Material for Use in Ceramic Gas Path Seal Systems

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United Technologies Research Center East Hartford, CT 06108

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Lewis Research Center Cleveland, Ohio 44135



# UNITED TECHNOLOGIES RESEARCH CENTER



East Hartford, Connecticut 06108

NASA CR-165469

Development of Low Modulus Material For Use in Ceramic Gas Path Seal Systems

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R. C. Novak

Final Report

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#### I. INTRODUCTION

Current turbine outer gas path seal systems based on metallic designs have two prime disadvantages related to performance: excessive cooling requirements and large turbine blade tip clearance. It is necessary to bleed air from the rear section of the compresser of the engine for cooling of metal turbine blade tip seals to maintain them within safe operating temperature limits. This cooling results in an engine performance penalty due to loss of high pressure air from the cycle. The injection of this air into the hot gas flow path also reduces turbine efficiency. Inadvertent rubbing between blade tips and the static seal shroud occurs due to seal shroud distortions, thermal and dynamic transients, and rotor bending when operating with tight clearances. Since present turbine seals for the most part are not abradable, a conservative approach is taken in establishing turbine operating alearnaces to prevent rotor wear. This promotes leakage of high energy air over the blade tips without work being extracted by the blades, thus lowering the turbine efficiency.

Because of these disadvantages associated with metallic systems, ceramic outer gas path seals are under development currently by both industry and government for use in high performance commercial and military aircraft engines. The ceramic seal concept offers two important features which make the system very attractive. First, higher engine operating temperatures are achievable because of the high temperature capability of ceramics coupled with their low thermal conductivity. Second, the ceramic seals can be made to be abradable which leads to improved gas path sealing.

To realize the advantages of ceramic seals, a design must be developed that solves the problem arising from the difference in thermal expansion coefficients between the cobalt or nickel based substrates presently in use and the ceramic layer. This difference can be sufficient to lead to bonding failure in the region of the ceramic-substrate interface.

Presently, two approaches can provide a solution and both are under development. One approach (Ref. 1) involves plasma spraying sequentially graded composition intermediate layers between the substrate and ceramic along with controlling the stress state of the layers. Stress control is necessary since plasma sprayed ceramic/metal systems can fail in a brittle fashion and preloading is seen as a means to increase the durability of the seal system. The other (Ref. 2) involves a low modulus strain isolator cushion between the substrate and ceramic wherein differences in thermal expansion are accommodated by deformation of the low modulus layer.

The objective of this program was to develop and characterize low modulus material systems capable of successfully performing in a seal application at a material temperature of at least  $1255\,^{\circ}\mathrm{K}$ . The approach involved the evaluation and optimization of three concepts. These are: Brunsbond (R) Pad, plasma sprayed porous metal, and plasma sprayed low modulus ceramic.

The program was divided into four technical tasks. The candidate systems were chosen in Task I. Task II involved the screening of variations of each system. Task III involved detailed property measurements. Task IV involved further investigation of a selected system.

#### II. TASK I - SELECTION OF CANDIDATE MATERIAL SYSTEMS

#### 2.1 Criteria for Candidate Low Modulus Strain Isolator Systems

The low modulus, strain-isolator interlayer between the metal substrate and ceramic surface layer is the key building block of the ceramic outer gas path seal system.

As a whole the seal system must be designed to withstand the demands of the operating environment. For the interlayer, these demands require that the structural integrity be sufficient for exposure to operational conditions. The interlayer must be capable of withstanding thousands of stress cycles induced by thermal gradients due to engine start-up, shut-down and operation. This is one of the most critical and most difficult requirements to meet. The design of the bonding between the high temperature ceramic surface layer and the metal substrate structure is critical to the success of the seal system. The interlayer must successfully absorb differential thermal expansion and maintain the strains in the ceramic surface layer within allowable limits for all foreseeable thermal cycles. Finally, the interlayer must satisfactorily withstand imposed forces due to vibration, pressure differentials, blade tip rub, hard landings, etc.

The physical properties to define and characterize the strain isolator interlayer include: (1) density, (2) chemistry, and (3) microstructure. These properties provide the basis for control of the system and the basis for the establishment of relationships between the other physical, mechanical, and thermal properties.

The thermal properties which are necessary to determine the temperature and strain distribution throughout the seal system are: (1) thermal conductivity, (2) specific heat, (3) density, and (4) thermal expansion. These properties are critical because the primary source of stress in the low modulus system results from differences in the thermal expansion and differences in temperatures of the ceramic and the metal substrate.

The mechanical properties which are required to determine stress levels within the low modulus system are: (1) modulus of elasticity, (2) strength, and (3) strain capacity. The modulus of elasticity is required to translate the calculated thermal strains into thermal stresses. The strength and strain capabilities of the material are used to define safe limits of operation.

As a result, the NASA selection criteria for this program included the following:

- . Potential to attain modulus of elasticity in the range of 3.5 to 6.9 GPa (.5 to 1 MSI), and ultimate strength of 17.2 MPa (2.5 KSI)
- . High thermal conductivity

- . Static oxidation life of at least 1000 hours at 1311°K.
- . Feasibility of attaching the low modulus system to a broad range of substrate alloys
- . Feasibility of depositing or forming the low modulus system to thicknesses of  $0.2~\rm cm$  to  $0.4~\rm cm$  (.075" to .150")

Three candidate systems were chosen for evaluation. First the fiber metal Brunsbond<sup>R</sup> Pad concept served as the baseline for the program. The second candidate involved plasma spraying a porous metal layer directly onto a prepared metal substrate. This offered the possibility of more simple fabrication, lower costs, and greater ease of refurbishment than the first approach. The third candidate involved a low modulus ceramic layer plasma sprayed directly onto a prepared substrate.

These approaches, the rationale for proposing them, and the evaluation techniques are discussed more fully in the following four sections.

#### 2.2 Fiber Metal Concept

The low modulus strain isolator approach requires a material that has a lower elastic modulus than the ceramic top layer and metal substrate to allow the ceramic and metal to strain independently of each other to minimize stresses within the structure. A low modulus fiber metal material is commercially available from Brunswick Corporation and possesses the candidate elastic and strength properties (Ref. 3).

Brunswick produces a fiber metal product consisting of 0.142 mm diameter Hoskins 875 fibers in pad form, marketed as Brunsbond<sup>R</sup> Pad. This is available in different densities and thicknesses. Attachment of the pad to a substrate is accomplished by a metallurgical braze process such as with a Nicrobraze foil. A ceramic top layer can then be plasma sprayed directly onto the bondcoated fiber pad surface. Brunsbond<sup>R</sup> Pad is being evaluated by Pratt and Whitney Aircraft-Government Products Division (Ref. 4), by NASA (Ref. 2), and by Brunswick (Ref. 3) for ceramic gas path seal use.

Brunsbond<sup>R</sup> Pad at 35% true density and 2.54 mm thickness was chosen for evaluation as the baseline system in this program. Specimens of 30% and 40% density and 2.54 mm thickness were also considered for possible lower modulus or increased thermal conductivity.

#### 2.3 Plasma Sprayed Porous Metal

The ability of the fiber metal approach to produce a low modulus material is in part due to the attenuation of stiffness as a result of the incorporation of porosity into the metal structure. Another approach to produce a low modulus cushion

between the ceramic seal and the metal substrate is to plasma spray a porous metal layer. This approach is very attractive for two reasons. First, plasma spraying technology provides the means to fabricate coatings in a cost and labor efficient manner. Second, plasma spraying technology is under active development as a method to fabricate ceramic seal systems, and consequently, a plasma sprayed low modulus cushion could be integrated readily into a continuous spray process.

Certain requirements must be addressed in considering the fabrication of a plasma sprayed, low modulus, porous metal cushion. These include attachment of the cushion to a wide variety of substrate alloys and the achievement of specified oxidation resistance, and strength, modulus, and coaling thickness values. Methods of addressing these requirements within the plasma sprayed porous metal approach are described briefly below.

#### 2.3.1 Attachment Requirements

To attach the plasma sprayed porous coating to substrates of low carbon steel, stainless steel, titanium, and aluminum, a 5 mil thick bond coat should be used, such as a blend of 94 Nichrome/6 aluminum (Metco 443). Metco literature (Ref. 5) values indicate that bond strengths to these substrates are of the order of 4000 psi. Research Center experience indicates that this bond coat material is also applicable to nickel and cobalt based superalloys.

#### 2.3.2 Oxidation Resistance Requirements

Alloys that form an alumina scale on exposure to air at elevated temperatures usually exhibit excellent oxidation properties. The compounds such as NiAl, Ni<sub>3</sub>Al, and the materials that are grouped together as MCrAlY, where M is either iron, nickel, or cobalt, fall into this category. In general, the chromium content of MCrAlY varies from about 12 to 25 w/o and the aluminum level is within the range of 4 to 13 w/o. Yttrium at a level of approximately 0.5 w/o is added to enhance scale adherence.

Experience at the Research Center and at NASA (Ref. 6) indicated that the NiCrAlY would be a suitable alloy. The composition chosen for this program was Ni-25Cr-6Al-.36Y.

#### 2.3.3 Property Requirements

Strength and modulus values of porous metal can be affected by controlling the porosity through the incorporation of a fugitive constituent such as polyester. To control the strength and modulus of the porous NiCrAlY, three levels of polyester (Metco 600) were blended with NiCrAlY and sprayed under Task II.

#### 2.4 Plasma Sprayed Low Modulus Ceramic

The fiber metal and porous metal approaches to fabricate a low modulus cushion involve metal systems. The third approach involves the use of a plasma sprayed low modulus ceramic loyer. A low modulus ceramic layer offers a distinct advantage over the preceding two systems in the area of high temperature oxidation resistance.

Figure 2-1 shows a typical 4 pt bend stress versus strain curve for a plasma sprayed ceramic material of low modulus in which the loading of the system is kept below the fracture strength level. As indicated, the system exhibits both elastic and inelastic behavior as non-recoverable deformation. This behavior is observed also with compression testing. Inelastic behavior at room temperative is atypical of ceramic materials and is the result of the plasma spray process. Sprayed ceramics exhibit markedly different strength and modulus values in addition to distinct microstructures depending on the plasma spray parameters used in fabrication. Low strength-low modulus specimens exhibit both porosity and splat morphology. The loose, mechanically interlocked microstructure is responsible in part for the observed nonlinear behavior in the stress-strain curve. Also, cracks that occur under stressing can be deflected and effectively arrested by the splat morphology. Because of this nonlinear behavior of the plasma sprayed ceramic materials, this approach offered promise, although not without risk, of successfully producing a low modulus plasma sprayed ceramic cushion for this application.

There are at least three possible techniques to influence the strength, modulus, and compliance of a plasma sprayed ceramic. These include adjustment of the plasma spray parameters to affect the microstructure; direct incorporation of porosity through the use of a fugitive such as polyester; and the development of microcracks in the ceramic as the result of the fabrication process. All three techniques were evaluated in Task II with yttria stabilized zirconia (Metco 202 NS) as the ceramic.

#### 2.5 Evaluation Procedures

Evaluation testing was performed on each candidate to guage its ability to perform in a strain isolator configuration. In Task II, data was obtained on the strength and oxidation properties of each. In Tasks III and IV, strength, static and cyclic oxidation behavior, and thermal properties were measured. The microstructure was examined and attachment to a substrate was demonstrated. Machining and testing procedures are described below.

#### 2.5.1 Machining

Special machining techniques are necessary when dealing with porous metals or ceramics because these materials are very susceptible to chipping or cracking during the machining operation. In order to avoid these problems, samples are impregnated with either beeswax or stickwax prior to machining. After the specimens are machined to the desired shapes and dimensions, the wax is removed by dissolution

in either tricholoroethylene or methanol, depending upon the type of wax which is used. To insure the removal of small traces of wax that may remain, the specimens are refluxed in the appropriate solvent. For surface finishing, a 320 grit grinding wheel is used.

#### 2.5.2 Tensile Properties

Both room temperature and 1089°K mechanical property tests were performed on samples in both the as-fabricated condition and after static exposure to 1311°K in air for 500 hours. The modulus of elasticity, ultimate tensile strength, and strain to failure were determined for each specimen.

In instances where gripping difficulties were encountered, the specimens underwent 4-point bend tests rather than true tensile tests. For the room temperature tests the strain was determined using a strain gage, and for the elevated temperature tests the strain was determined by use of an extensometer.

#### 2.5.3 Oxidation Testing

Static oxidation testing was carried out at 1311°K in a still air resistance furnace for a duration of 500 hours. Weight gain and dimensional change measurements were made after 1, 10, 100, 200 and 500 hours.

Cyclic oxidation tests were carried out in a specially-made rig which allows for sample movement in and out of a resistance heated tube furnace. The specimens were held at a maximum temperature of 1311°K for 65 minutes and then raised out of the furnace and air cooled for 15 minutes. The total accumulated time at 1311°K was one-hundred (100) hours. Weight gain and dimensional change measurements were made after accumulated times of 1, 10, 50 and 100 hours at 1311°K.

#### 2.5.4 Thermal Conductivity

The thermal conductivity of each material was determined by the Thermophysical Properties Research Laboratory of Purdue University. Each material was tested from room temperature to 1089°K in the thickness direction. The density, specific heat, and thermal diffusivity were measured and the thermal conductivity calculated from these results.

The bulk densities were determined from the mass and geometries of the samples. The specific heat values were measured with a differential scanning calorimeter. Thermal diffusivity values were determined using the laser flash technique.

As described in TPRL Report # 249, the laser flash apparatus consists of a Korad K2 laser, a high vacuum system including a bell jar with windows for viewing the sample, a tantalum tube heater surrounding a sample holding assembly, an IR detector, appropriate biasing circuits, amplifiers, A-D converters, crystal clocks,

and a minicomputer-based digital data acquisition system capable of accurately taking data in the 40 microsecond and longer time domain. In the apparatus a small disc shaped sample of the candidate material is subjected to a short laser burst and the resulting rear face temperature rise is recorded. The computer controls the experiment, collects the data, calculates the results, and compares the new data with the theoretical model.

#### 2.5.5 Microstructural Investigation

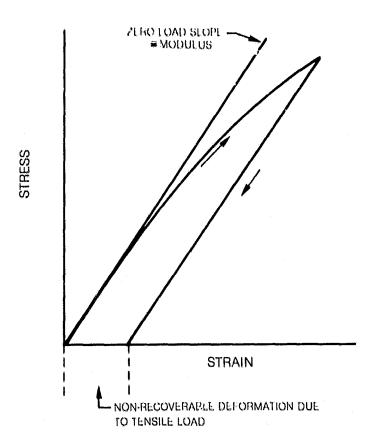
The as-fabricated specimens were subjected to metallographic examination in order to determine important microstructural features. The specimens which underwent static oxidation tests and cyclic oxidation tests were also examined.

#### 2.5.6 Attachment Demonstration

Each of the candidate systems was attached to stainless steel and cobalt based alloys to demonstrate attachment feasibility. For the fiber pad, a brazing process was employed, while for the sprayed systems Metco 443 bondcoat was used.

## TYPICAL STRESS VS. STRAIN CURVE FOR A PLASMA SPRAYED CERAMIC OF LOW STRENGTH

(MAXIMUM LOAD & FRACTURE STRENGTH)



#### III. TASK II - PRELIMINARY EVALUATION OF SELECTED SYTEMS

Figure 3-1 summarizes the systems along with variations in these systems selected for preliminary evaluation in Task II. The preliminary evaluation conducted in Task II consisted of room temperature mechanical property tests, and static oxidation at 1311°K for 24 hours.

#### 3.1 Fiber Metal Approach

Specimens of Brunsbond<sup>R</sup> Pad were purchased from Brunswick Corp., Deland, FL. The Brunsbond<sup>R</sup> Pads were composed of 0.142 mm fibers of Hoskins 875 alloy sintered together to form different densities. Nominal densities of 30%, 35% and 40% were evaluated.

Mechanical property specimen dimensions were 12.7 cm x 2.54 cm x 0.254 cm. Fiberglass doublers, 4.0 cm in length, were epoxied to the ends of each specimen. The gage length between doublers was 4.7 cm, and attached to the specimen in this region was a 2.54 cm extensometer for strain measurement. Figure 3-2 summarizes the average mechanical properties versus density for the Brunsbond  $^{\rm R}$  Pads. The actual data are presented in Table A-1.

For these tensile tests, failure generally occurred outside the gage length of the extensometer, and, as a result, strain to failure was reported in most cases as being greater than 1%. In subsequent tensile tests, each doubler component was tapered to promote failure within the gage length.

Static oxidation of the three densities for 24 hours at  $1311^\circ K$  produced less than 0.6% weight gain as indicated in Table A-2.

#### 3.2 Porous NiCrAlY

Specimens of perous NiCrAlY at three different densities (4780, 2870 and 2390  $\rm Kg/M^3$ ) were fabricated by plasma spraying blends of polyester and NiCrAlY. The polyester was Metco 600 and the NiCrAlY was Ni-25Cr-6Al-.35Y initially sieved to -170+325 mesh, received from Homogeneous Metals, Inc., Clayville, New York. The spray parameters and powder blends are described in Table A-3.

Four point bend mechanical property test specimens of each density were machined out of sprayed monoliths having dimensions of approximately 5 cm  $\times$  10 cm  $\times$  .37 cm. Test specimens were 2.75 cm  $\times$  1.0 cm  $\times$  .25 cm. Prior to mechanical property or oxidation testing, the polyester was baked cut by exposure to 810°K for 1 hour.

Figure 3-3 summarizes the mechanical properties versus density for the porous NiCrAly. The data are presented in Table A-4. Strain was measured by both a

deflectometer and a strain gage. The diffurence between strain gage and deflectometer modulus values can be attributed either to machine compliance at low loading for the deflectometer values or to infiltration of the porous NiCrAlY by the strain gage epoxy. The true modulus value probably lies between the two reported values.

Static oxidation is summarized in Fig. 3-4 and the data are presented in Table A-5. Oxidation measurements based on percent weight gain reflect the high surface area of a plasma sprayed porous metal system.

#### 3.3 Low Modulus Ceramic

Specimens of low modulus zirconia were produced having three distinct microstructures. A porous microstructure, Fig. 3-5, was produced by plasma spraying a blend of Metco 600 polyester and Metco 202 NS zirconia. A microcracked microstructure was produced by plasma spraying Metco 202 NS under conditions that produced a controlled amount of microcracking (Fig. 3-6). Work at the Research Center has shown that microcracks can be produced in plasma sprayed zirconia through the control of certain spray parameters such as voltage, amperage, injector configuration and substrate cooling. It is believed that the specimen is thermally shocked continuously during spraying resulting in the microcrack network. In the third technique, a loosely bound microstructure, Fig. 3-7, was produced by plasma spraying Metco 202 NS zirconia with spray parameters selected to accomplish this. In each case monoliths were sprayed having dimensions of approximately 5 cm x 10 cm x .37 cm. From these monoliths, individual 4 point bend specimens were cut having dimensions of 2.75 cm x 1 cm x .25 cm.

Figure 3-8 summarizes the 4-point bend strength and modulus values of these systems. The data are presented in Table A-6.

Static oxidation at 1311°K for 24 hours shows no weight gain. The data are presented in Table  $\lambda$ -7.

#### 3.4 Selection of Task III Systems

Based on testing performed under Task II and in conjunction with discussions between the Research Center and the NASA Program Manager, the following three systems were selected for evaluation in Task III.

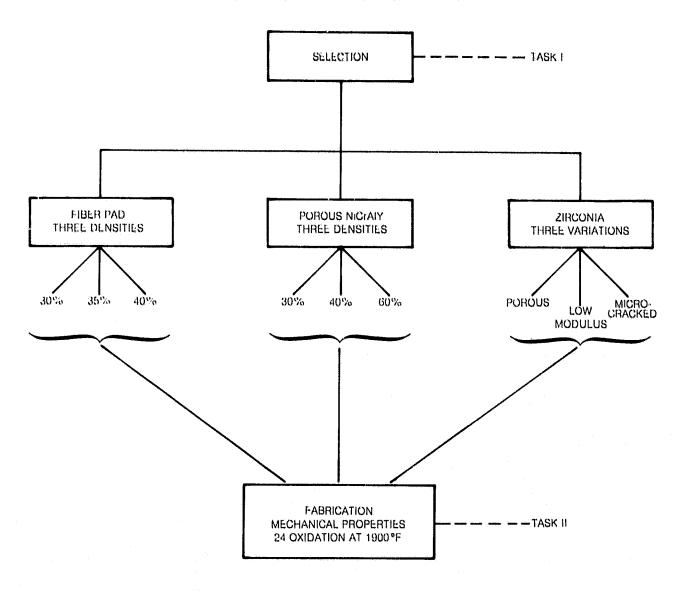
- . Brunsbond<sup>R</sup> Pad at 35% density
- . Porous NiCrAlY at 2870 kg/M<sup>3</sup> density
- . Microcracked zirconia

Brunsbond<sup>R</sup> Pad at 35% density represented a baseline strain isolator system (Ref. 4). Increased density leads to increased modulus while decreased density is not optimum from a strength viewpoint.

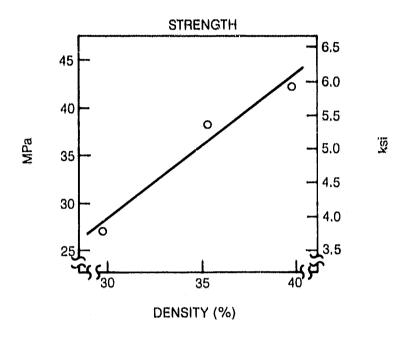
Porous NiCrAlY at 2870 kg/M<sup>3</sup> density represented the best choice based on strength and modulus data. In an attempt to reduce oxidation, a coarser grade of NiCrAlY powder was evaluated early on in Task III.

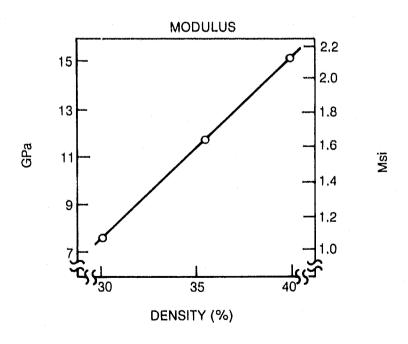
Microcracked zirconia represented an interesting system since the cracks could be controlled and were oriented in a fashion which might accommodate the differences in thermal expansion between the seal substrate and top layer.

## CANDIDATE LOW MODULUS STRAIN ISOLATOR SYSTEMS AND VARIATIONS SELECTED IN TASK I

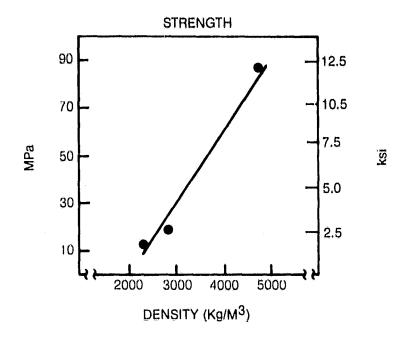


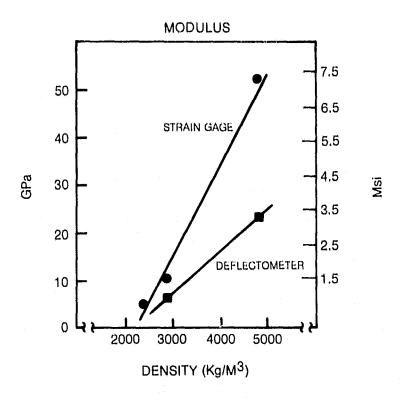
## ROOM TEMPERATURE TENSILE PROPERTIES VS DENSITY OF BRUNSBOND® PAD





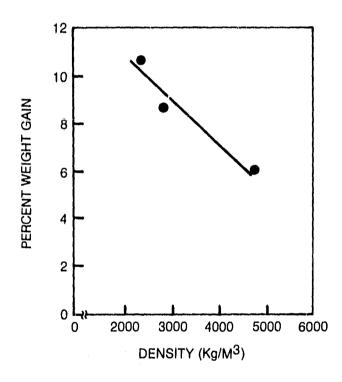
## ROOM TEMPERATURE FOUR POINT BEND PROPERTIES OF POROUS NICIALY VS DENSITY





## WEIGHT PERCENT OXIDATION POROUS NICIAIY VS DENSITY AT 1310°K FOR 24 HOURS

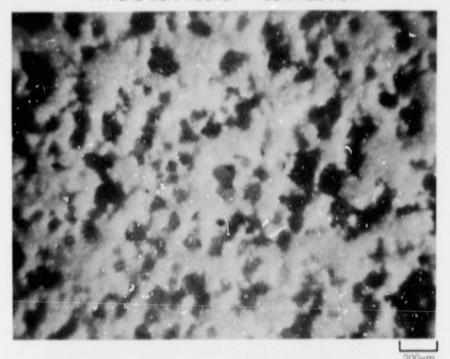
(-170 + 325 MESH NICRAIY)



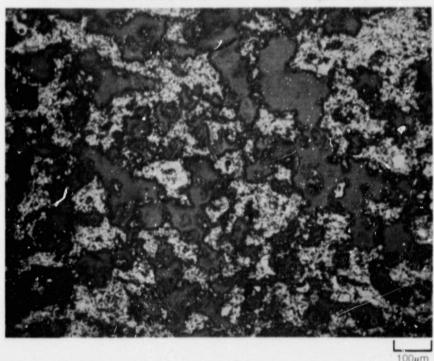
## PLASMA SPRAYED POROUS ZIRCONIA

LIGHT REGIONS = ZrO2 DARK REGIONS = POROSITY

#### TYPICAL MORPHOLOGY — SURFACE VIEW

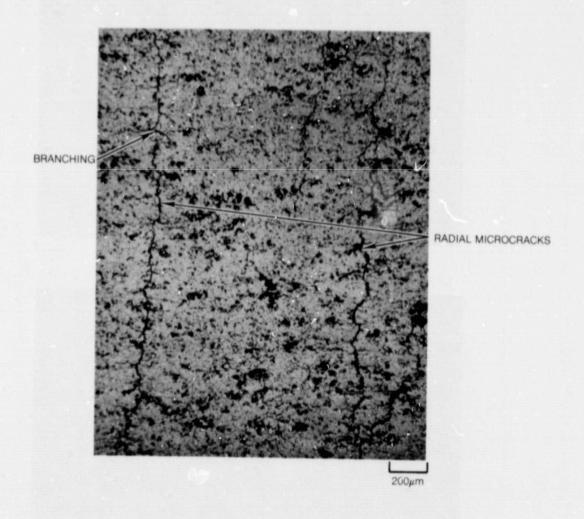


UTRC NO. 80-95 POROUS ZIRCONIA — CROSS SECTIONAL VIEW



## PLASMA SPRAYED MICROCRACKED ZIRCONIA

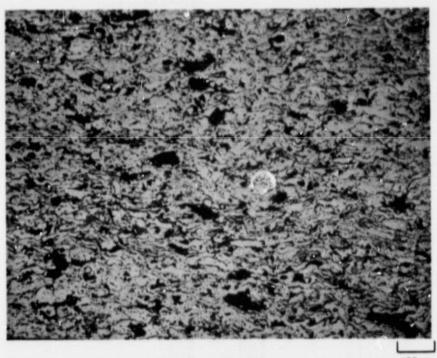
UTRC NO. 80-96



## PLASMA SPRAYED LOOSELY BOUND ZIRCONIA

UTRC NO. 80-98

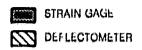
NOTE SPLAT OUTLINES OF ZIRCONIA PARTICLES

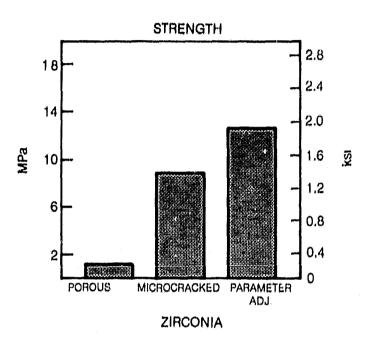


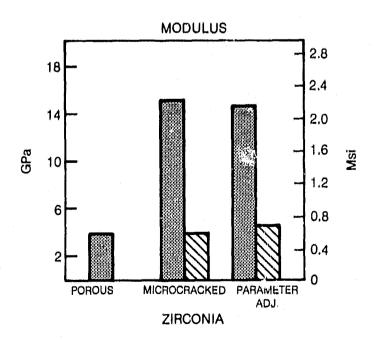
100µm

OF POOR QUALITY

## FOUR POINT BEND PROPERTIES OF LOW MODULUS ZIRCONIA SPECIMENS







#### IV. TASK III - EVALUATION OF CANDIDATE SYSTEMS

Figure 4-1 summarizes the systems selected in Task II for evaluation in Task III. For Task III, a coarser fraction of NiCrAly was selected in an effort to reduce oxidation. For this effort, -100 + 200 mesh NiCrAly was chosen and used throughout the remainder of the program. Evaluation in Task III consisted of the following asting.

- . Measurement of strength and modulus at room temperature and at 1089°K.
  This was accomplished both in the as fabricated condition and after aging at 1311°K for 500 hours.
- . Static oxidation at 1311°K for periods of 1, 10, 100, 200, and 500 hours. Weight and dimensional changes were monitored for each interval.
- . Cyclic oxidation between room temperature and 1311°K. Each cycle consisted of 65 minutes at 1900°F and 15 minutes at room temperature. Weight and dimensional changes were recorded after 1, 10, 50 and 100 cycles.
- . Thermal conductivity. Measurements were made in the thickness direction at temperatures between room temperature and 1116°K. Measurements were made by Purdue University.
- . Attachment. Demonstration was accomplished by attaching each system to both a 304 SS substrate and to a Haynes-25 substrate.
- . Optical microscopy. Photomicrographs were obtained to examine the as fabricated state of each system and the effect of thermal aging.

#### 4.1 Mechanical, Oxidation, and Thermal Tests

#### 4.1.1 Results

Figure 4-2 compares strength and modulus values of the three systems in the as fabricated and aged 500 hours at  $1311^{\circ}$ K conditions. The data are presented in Tables A-8, A-9, A-10, A-11, A-12 and A-13. For the Brunsbond<sup>R</sup> Pad, failure occurred within the gage length of the extensometer, indicating that the tapered doublers were an improvement over the non-tapered configuration of Task II.

Figure 4-3 compares static and cyclic oxidation of the three systems. Dimension changes and oxidation data are presented in Tables A-14, A-15, A-16, A-17, A-18 and A-19.

Figures 4-4, 4-5 and 4-6 compare specific heat, thermal diffusivity, and thermal conductivity as functions of temperature for the three systems. The data are presented in Tables A-20, A-21 and A-22. This work was performed by R. E. Taylor and H. Groot, TPRL 249, Thermophysical Properties Research Laboratory, School of Mechanical Engineering, Purdue University, West Lafayette, Indiana.

#### 4.1.2 Discussion

## 4.1.2.1 35% Dense Brunsbond<sup>R</sup> Pad

- 1. Modulus remains between 10.3 and 3.45 GPa (1.5 and 0.5 MSI) at temperatures between RT and 1089°K. This is true even after extensive exposure to severe static oxidation conditions (500 hours @ 1311°K), as shown in Fig. 4-2.
- 2. Questions arise concerning the high temperature structural behavior. Linear extrapolation of the two strength values (Fig. 4-2) indicates that the strength goes to a low value at the upper temperature range of interest (1255-1311°K). While an extrapolation must be recognized for what it is, the high temperature strength of Brunsbond<sup>R</sup> Pad should be examined further.

In particular, the following questions should be addressed:

- . What is the relationship between strength of the fiber pad and Hoskins 875 alloy from which the fibers are fabricated? Is the fiber pad strength simply a ratio of the alloy strength?
- . What is the low cycle fatigue behavior as a function of temperature?
- . How does entanglement of the fibers affect the measured strength, especially at high temperature?
- . What are the high temperature creep properties of the material? If the creep resistance is low, the pad may truly be a strain isolator under steady state conditions.
- What is the effect of crosshead speed on property measurements? Data presented here was obtained at 0.254 mm/min strain rate. Literature data (Ref. 7) gives a 1089°K strength of 1000 psi for 35% dense Brunsbond<sup>R</sup> Pad which was produced at a crosshead speed of 5.09 mm/min (Ref. 8).
- 3. Static and cyclic oxidation (summarized in Fig. 4-3) show comparable weight and dimensional changes. A total of +1.3% increase in weight occurred after 500 hours at  $1311^{\circ}$ K in static oxidation and no more than one percent dimensional change occurred under these conditions.

- 4. Thermal conductivity ranges from  $\sim 0.01~\rm W~cm^{-1}K^{-1}$  at room temperature to 0.026 W cm<sup>-1</sup>K<sup>-1</sup> at 1116°K. The peak in the specific heat versus temperature curve at  $\sim 800$ °K is associated with the Curie point. Specimens used for this test were .254 cm thick and brazed (by Brunswick) to a stainless steel substrate. Thickness of the fiber metal might have an effect on the measured thermal conductivity value. This could occur due to heat transmission through the Brunsbond Pad by radiation since the pad is of low density and thin specimens are optically transparent in part.
- 5. Microstructurally (Fig. 4-7), strength at room and elevated temperature appears to be due to the amount of bridging between the fibers and the fiber entanglement. Oxidation is limited to oxide formation at the fiber surface (Fig. 4-8). No internal oxidation is indicated after 500 hours at 1900°F since porosity within the fibers is absent. Brunsbond Pad at 35% density has a potential life of 1000 hours at 1311°K.

#### 4.1.2.2 Porous NiCrAlY

1. Modulus of the as-fabricated material (Fig. 4-2) is between 9.6 and 6.9 GPa (1.4 and 1.0 MSI) between room temperature and 1089°K. After static oxidation for 500 hours at 1311°K, the modulus is 23.4 to 18.6 GPa (3.4 to 2.7 MSI) between room temperature and 1089°K.

The increase in modulus with thermal aging is probably due to sintering and the conversion of metal to oxide.

- 2. As-fabricated strength is 26.1 to 18.6 MPa (3.8 to 2.7 KSI) between room temperature and 1089°K. Aged strength is 48.2 to 20.0 MPa (7.0 to 5.8 KSI) between room temperature and 1089°K. The increase in strength with thermal aging is consistent with the sintering and formation of oxide products.
- 3. Static and cyclic oxidation produce similar results. Static oxidation, Fig. 4-3, after 500 hours at 1311°K gives a weight gain of + 10.5%. There is less than +2% change in dimension after 500 hours at 1311°K. The measured weight gains with oxidation are consistent with a high specific surface area plasma sprayed metal. Dimensional changes due to oxidation could give rise to stresses at the ceramic-porous NiCrAlY interface and this could limit the high temperature use.

Oxidation can cause problems because conversion of NiCrAly metal to oxides should occur at the ceramic NiCrAly interface. This is likely because of the temperature at this position and the high oxygen potential especially with a zirconia layer due to its oxygen ion conduction capability. Spalling of the oxide scale could occur due to thermal cycling leading to failure of the system along the interface. Means to limit oxide formation at the interfacial region should be investigated and could include the following:

- . Vapor deposition of a protective coating.
- . Argon atmosphere spraying of the NiCrAlY to promote metallurgical bonding between the splats to decrease effective surface area.
- . Oxidation of the sprayed layer prior to ceramic deposition.
- 4. Thermal conductivity, Figs. 4-4, 4-5, & 4-6, ranges from  $\sim 0.007$  W cm<sup>-1</sup>K<sup>-1</sup> at room temperature to  $\sim 0.02$  W cm<sup>-1</sup>K<sup>-1</sup> at 1116°K.
- 5. Microstructurally (Fig. 4-9), plasma sprayed porous NiCrAlY has a very high surface area. This produces high measured oxidation values due to the formation of oxides around each splat (Fig. 4-10). Means to reduce oxidation should be centered around reducing the specific surface area or reducing exposure to temperature. Oxide formation should produce a decrease in the coefficient of thermal expansion which may offset an increase in modulus. Since oxidation occurs primarily in the hottest region of the low modulus interlayer, i.e., along the ZrO<sub>2</sub>/NiCrAlY interface, the cooler region should maintain its modulus.

#### 4.1.2.3 Microcracked Zirconia

- 1. Modulus, Fig. 4-2, is 6.2 to 3.4 GPa (.9 to .5 MSI) between room temperature and 1089°K for the as-fabricated condition, and from 17.2 to 10.3 GPa (2.5 to 1.5 MSI) in the aged condition for that temperature range.
- 2. Strength measurements show 9.6 to 11.7 MPa (1.4 KSI to 1.7 KSI) between 70 to  $1089^{\circ}$ K for the as-fabricated condition and 11.0 to 14.5 MPa (1.6 to 2.1 KSI) for the aged.
- 3. Oxidation testing, Fig. 4-3, shows that no significant weight or dimensional change occurred.
- 4. Thermal conductivity, Fig. 4-6, remains almost constant at 0.01 W cm $^{-1}$ K $^{-1}$ between room temperature and 1116°K.
- 5. Microstructurally (Fig. 3-6) zirconia has radial microcracks which exhibit a high degree of branching in the X-Y plane.

Thermal cycling of a free standing body of microcracked zirconia does not appear to link up the microcracks, Fig. 4-11. However, the behavior of the system under constraint is unknown. For this reason, it is necessary to conduct thermal cycling of the microcracked zirconia in a strain isolator seal configuration to properly evaluate it. Preferably a cyclic thermal gradient would be imposed but even a constant temperature thermal cycle would provide useful information.

#### 4.2 Attachment Demonstration

Each of the systems was fabricated onto stainless steel and Haynes 25 alloy substrates. The Brunsbond<sup>R</sup> Pad was attached by use of a brazing operation. The microcracked zirconia and porous NiCrAly were plasma sprayed directly onto Metco 443 bondcoated substrate surfaces. Specimens of each of these systems attached to both types of substrates were delivered to the NASA Program Manger, R. C. Bill.

#### 4.3 Selection of Task IV Candidate Material

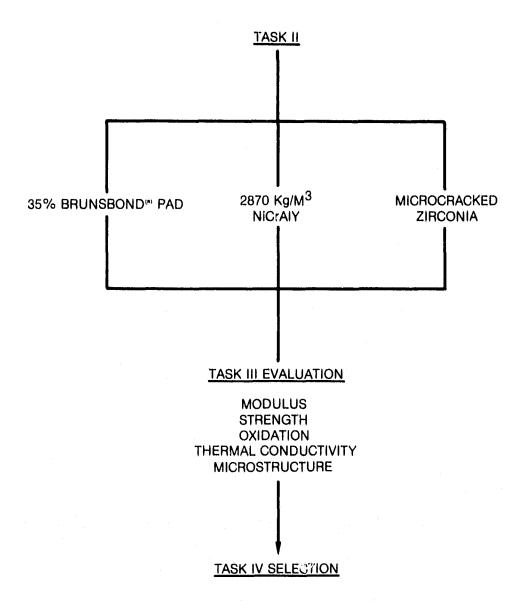
Each Task III candidate material offers attractive properties that warrant further testing.

The porous NiCrAlY system was selected for further evaluation in Task IV, based on discussions between UTRC and the NASA Program Manager. The NiCrAlY system can be used in any situation that the zirconia system can be used, and may offer fabrication advantages over the fiber metal approach.

The as-fabricated properties of porous NiCrAlY are desirable, however, the modulus increases with oxidation exposure. The amount of oxidation per time is largely due to the high specific surface area of the sprayed NiCrAlY. Efforts to decrease oxidation were made in Task II in switching from a -170 + 325 mesh to a -100 + 200 mesh powder. This produced roughly half the measured oxidation. A further increase in size is not practical from a spraying point of view.

In order to offset the increase in modulus with aging, a lower density (and therefore, lower modulus) NiCrAlY system was selected for evaluation in Task IV.

## TASK II SELECTED CANDIDATES AND TASK III EVALUATION

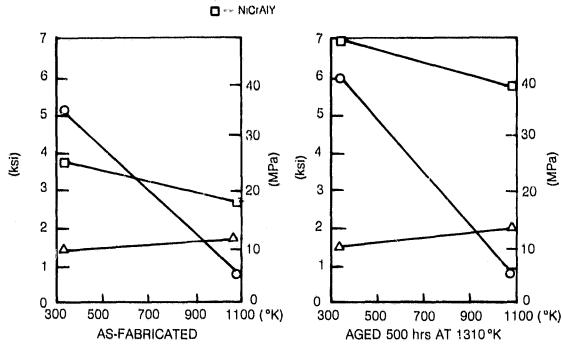


#### TASK III — SPECIMEN PROPERTIES

#### STRENGTH

▲ == ZIRCONIA

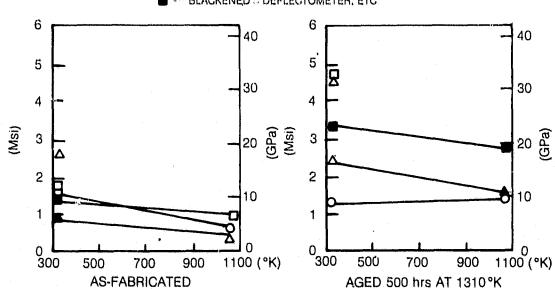
O - FIBERMETAL



#### **MODULUS**

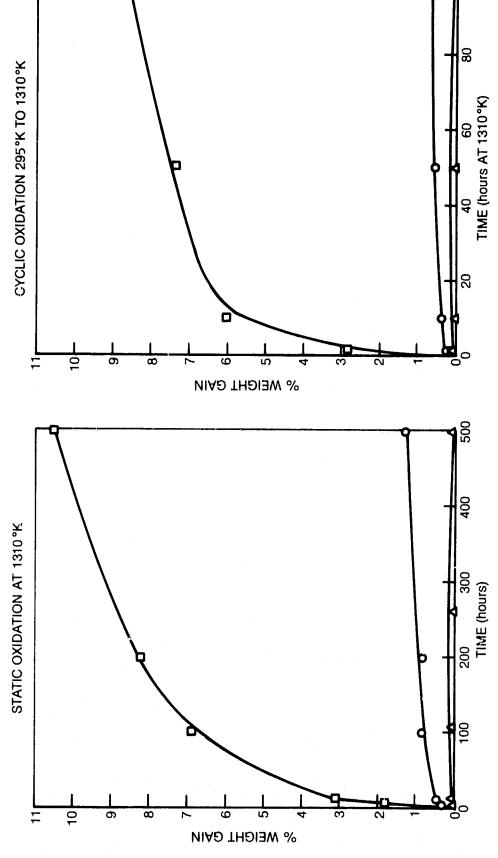
■ - OPEN - STRAIN GAGE

- BLACKENED = DEFLECTOMETER, ETC.



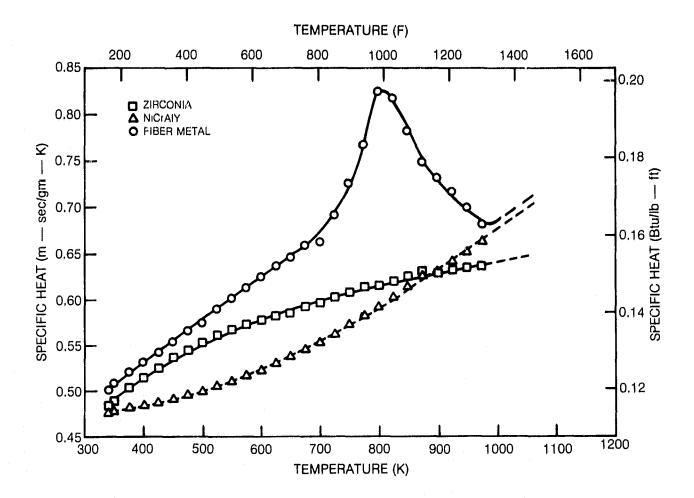
TASK III — SPECIMEN OXIDATION





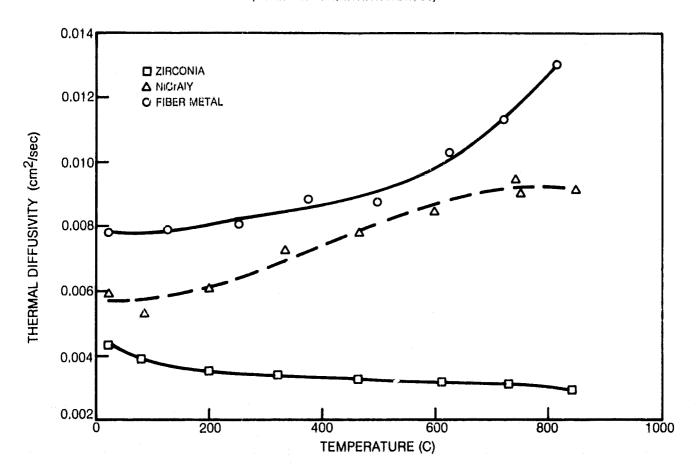
## SPECIFIC HEAT OF TASK III CANDIDATES

(FROM TPRL 249, TAYLOR & GROOT)



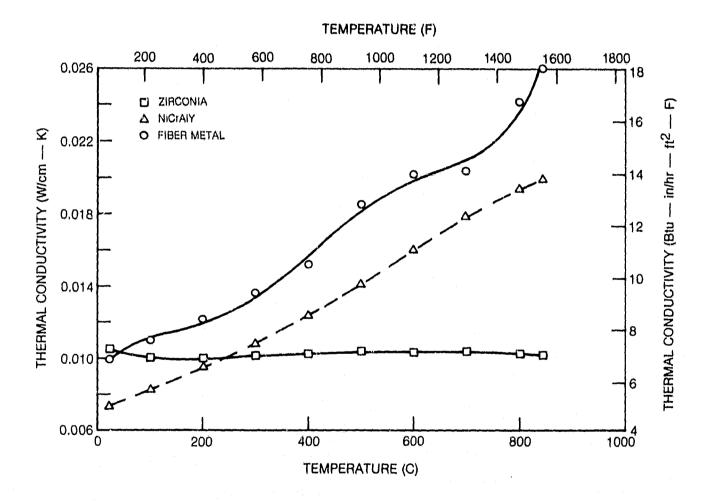
#### THERMAL DIFFUSIVITY OF TASK III CANDIDATES

(FROM TPRL 249, TAYLOR & GROOT)



#### THERMAL CONDUCTIVITY OF TASK III CANDIDATES

(FROM TPRL 249, 1AYLOR & GROOT)



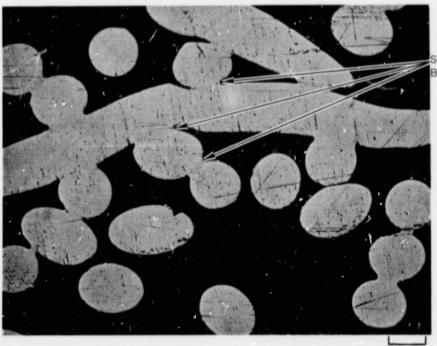
#### 35% DENSE BRUNSBOND® PAD

SURFACE VIEW SHOWING FIBER ENTANGLEMENT



700µm

CROSS SECTIONAL VIEW SHOWING BRIDGING BETWEEN FIBERS



SINTERED BRIDGES BETWEN FIBERS

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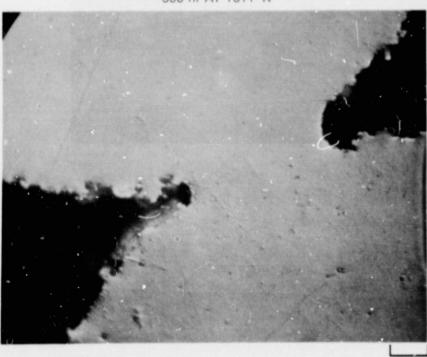
#### OXIDATION OF BRUNSBOND® PAD

#### AS RECIEVED



SMOOTH WIRE SURFACE IN AS FABRICATED CONDITION

500 hr AT 1311 °K



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SURFACE OXIDATION

81-8-92-16

#### PLASMA SPRAYED POROUS NICIAIY

UTRC NO. 81-16, DENSITY = 3010 Kg/M3

LIGHT AREAS = NICrAIY DARK AREAS = POROSITY

#### PLASMA SPRAYED POROUS NICIAIY EXHIBITS HIGH SPECIFIC SURFACE AREA



### PLASMA SPRAYED POROUS NICRAIY — TASK III OXIDE FORMATION ON THERMAL AGING





### MICROCRACKED ZIRCONIA AFTER THERMAI. CYCLING UTRC NO. 80-129

MICROCRACKS DO NOT APPEAR TO LINK UP DUE TO THERMAL CYCLING



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#### V. TASK TV - OPTIMIZATION AND EVALUATION OF A SELECTED CANDIDATE SYSTEM

Porous NiCrAly was chosen for further optimization in Task IV based on discussions between the Research Center and NASA Program Manager.

Task TIT data showed that the modulus of the porous NiCrAlY (density 3010 kg/M<sup>3</sup>) increased substantially with thermal aging. The room temperature modulus went from 9.7 GPa (1.4 MST) to 23.5 GPa (3.4 MST). At 1089 K the modulus increased similarly. This large increase in undesirable because it tends to defeat the concept of the low modulus strain isolator system.

Any technique limiting the amount of exidation in the perous NiCrAly would have a beneficial effect on maintaining the as-sprayed modulus. Decreasing the specific surface area of the structure is important and was achieved in Task III by using a more coarse fraction (-100 + 200 mesh) of NiCrAly powder than in Task II (-170 + 325 mesh). The result was an approximate halving of the measured exidation. Further increase in the powder size was not examined because it would probably decrease the efficiency of the spray fabrication process. Other possible means to effectively reduce the specific surface area (e.g., sputtering a protective coating or use of an argon spray atmosphere to promote metallurgical bonding) were not within the scope of this program.

The approach evaluated in Task IV to maintain an acceptable modulus in the porous NiCrAlY, involved starting with a lower modulus system. Specimens of porous NiCrAlY, density 2347 kg/M<sup>3</sup>, were prepared from -100 + 200 NiCrAlY powder and Meteo 600 polyester. The as-fabricated modulus was 2.96 GPa (.43 MSI) compared with 9.7 GPa (1.4 MSI) in Task III. With exidation, the modulus would increase but to a lower value than that obtained with the Task III system.

#### 5.1 Mechanical, Oxidation, and Thermal Tests

#### 5.1.1 Results

Figure 5-1 shows strength and modulus of the 2347 kg/M $^3$  porous NiGrAlY at room temperature and  $1089\,^{\circ}$ K in the as-sprayed and aged conditions. The data are presented in Tables A-23 and A-24.

Figure 5-2 shows static and cyclic exidation. The dimension change and exidation data are presented in Tables A-25 and A-26.

Figures 5-3, 5-4 and 5-5 show specific heat, thermal diffusivity, and thermal conductivity as functions of temperature for both the Task IV and Task III NiGrAlY systems. The data are presented in Tables A-27, A-28 and A-29. This work was performed by R. E. Taylor and H. Groot, TPRL 249A, Thermophysical Properties Research Laboratory, School of Mechanical Engineering, Purdue University, West Lafayette, IN.

#### 5.1.2 Discussion

- a. The modulus is between 2.96 and 1.77 GPa (0.43 and 0.26 MSI) for the as-fabricated condition between room temperature and 1089°K. After aging 500 hours at 1311°K, the modulus is between 11.8 and 11.5 GPa (1.71 and 1.67 MSI) for the same temperature range. This represents a substantial improvement over the Task III porous NiCrAly which exhibited an aged modulus range of 23.3 to 19.0 GPa (3.4 to 2.8 MSI).
- b. The strength is between 11.2 and 8.05 MPa (1.6 and 1.2 KSI) for the asfabricated condition between room temperature and 1089°K. After aging
  500 hours at 1311°K, the strength is between 20.4 and 12.2 MPa (2.97
  and 1.78 KSI). Compared with the Task III NiCrAlY (strength 26.4 to 18.6
  MPa as-sprayed and 48.3 to 39.8 MPa aged), the strength of the Task IV
  NiCrAlY has been reduced by the effort to preserve a desirable modulus
  in the aged condition. The decrease in strength is consistent with a lower
  density system.
- c. Static and cyclic oxidation summarized in Fig. 5-2 show similar effects of high specific surface area due to the high porosity of the Task IV NiCrAlY. After 500 hours at 1311°K, static oxidation results showed a 16% weight increase.
  - Calculations based on Ni-25Cr-6Al-.35Y indicate that a percent weight gain of about 17% would consume all the chromium, aluminum, and yttrium (assuming preferential oxidation of these elements and that  $\text{Cr}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Y}_2\text{O}_3$  are formed). The calculation is complicated by the different splat sizes present in the NiCrAlY structure such that nickel in the small splats would be consumed long before nickel in the large splats. However, an oxidation weight gain of +16% after 500 hours at 1311. K indicates that the fine structures within the NiCrAlY material probably have been converted totally to oxides while only the medium to large structures still exhibit a metallic phase. This is apparent in the microstructure (Fig. 5-6).
- d. Thermal conductivity of the Task IV NiCrAlY compared to the Task III NiCrAlY is related to the square of the ratio of the densities of the two. This can be seen from the following points: (1) the specific heat values are the same for the two specimens indicating that the material composition is the same; (2) the thermal diffusivity values of the Task IV NiCrAlY are reduced approximately by the ratio of the densities of the two systems; (3) finally, the conductivity is calculated by the formula  $K = C_p \alpha d$  where K is the conductivity,  $C_p$  is the specific heat,  $\alpha$  is the thermal diffusivity, and d is the density. Thus, the ratio of conductivities is related to the square of the ratio of densities. This fact is important because it indicates the dependency of conductivity on density for systems

at these levels. It may be possible to use this effect to advantage in the design of the system since a thin porous NiCrAlY layer of higher density at the interface with the ceramic should have improved oxidation resistance.

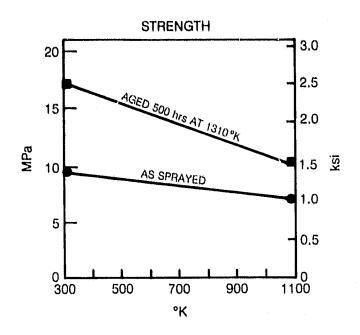
e. Microstructurally (Fig. 5-7), the Task TV NiCrAlY is similar to the Task III NiCrAlY but has a slightly lower density of metal.

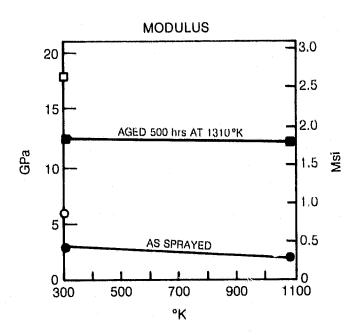
#### 5.2 Attachment Demonstration

The Task IV porous NiCrAlY, density 2347 kg/ $\rm M^3$ , was sprayed onto Haynes 25 alloy to demonstrate attachment. A Metco 443 bondcoat was used between the substrate and NiCrAlY strain isolator. A specimen of this system was delivered to the NASA Program Manager.

#### STRENGTH AND MODULUS OF 2347 Kg/M<sup>3</sup> POROUS NICTALY AT 295° AND 1310°F AS-SPRAYED AND AGED

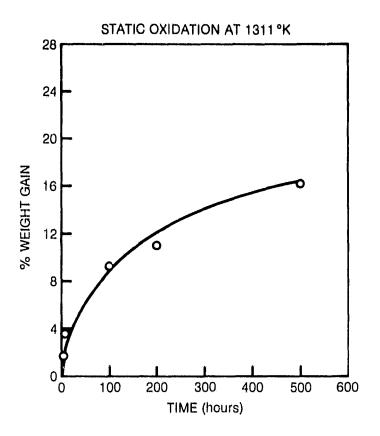
OPEN-STRAIN GAGE, EXTENSOMETER BLACKENED-DEFLECTOMETER

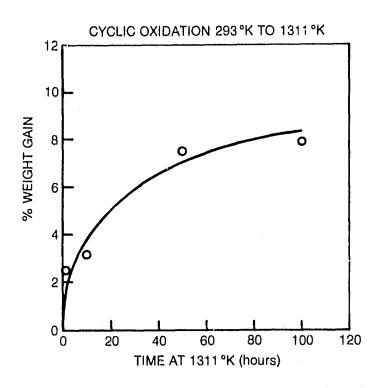




#### TASK IV NICRAIY OXIDATION

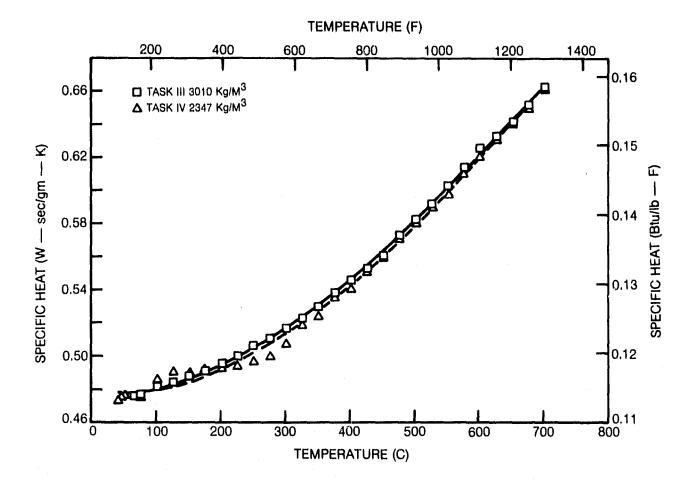
DENSITY 2347 Kg/M<sup>3</sup>





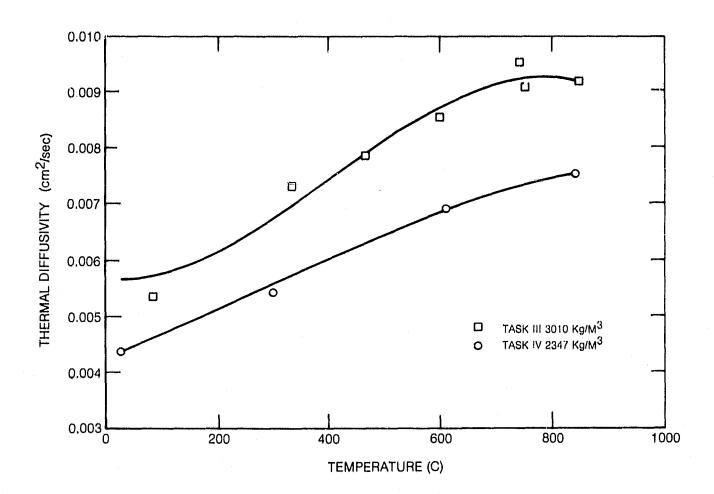
#### TASK IV NICIAIY - SPECIFIC HEAT

(FROM TPRL 249A, TAYLOR & GROOT)



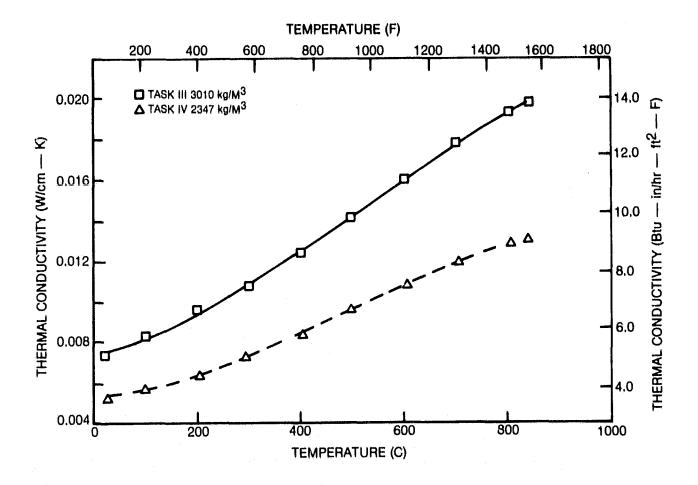
#### TASK IV NICIALY - THERMAL DIFFUSIVITY

(FROM TPRL 249A, TAYLOR & GROOT)



#### TASK IV NICIALY - THERMAL CONDUCTIVITY

(FROM TPRL 249A, TAYLOR & GROOT)



#### PLASMA SPRAYED NICIAIY

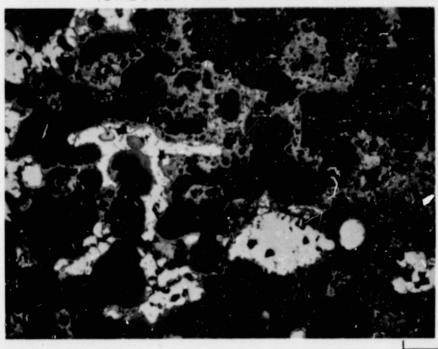
DENSITY = 2347 Kg/M3, AGED 500 hrs AT 1311 K

WHITE = METAL LIGHT GREY = OXIDE DARK GREY = MOUNTING MATERIAL

#### PARTIAL OXIDATION OF LARGE PARTICLES



TOTAL OXIDATION OF SMALL PARTICLES

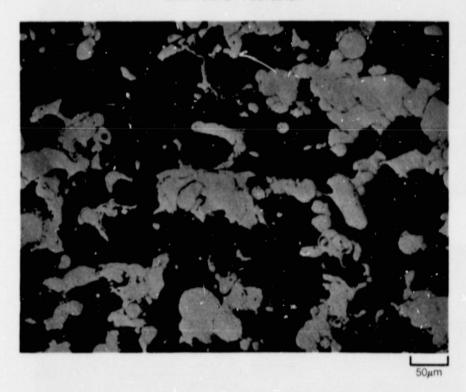


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m

#### PLASMA SPRAYED NICIAIY DENSITY 2347 Kg/M3 AS-FABRICATED

UTRC81-043 LIGHT AREAS = METAL DARK AREAS = POLYESTER



#### VI. CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

#### 6.1 Conclusions

- 1. All three canidate systems investigated in the program offer promise as low modulus interlayers, but each has features requiring further investigation.
- 2. Brunsbond Pad has a good combination of room temperature properties and oxidation resistance, but the brazing operation for attachment is a disadvantage, and further testing of high temperature properties appears necessary.
- 3. The plasma sprayed porous NiCrAlY system exhibits oxidation and any swelling of the material must be evaluated for its effect on performance.
- 4. The microcracked zirconia system is interesting and offers attractive features, especially in regards to oxidation. However, the system must be thermally cycled in a strain isolator configuration in order to properly assess whether the microcrack structure is functional in accommodating strain.

#### 6.2 Recommendations

- 1. Perform mechanical property tests on the systems of interest at the upper use temperature.
- 2. Measure the thermal expansion coefficients in the as-fabricated and aged conditions.
- 3. Examine the fatigue and creep properties of 35% dense Brunsbond  $^{R}$  Pad at room and elevated temperatures.
- 4. Examine methods to decrease the effect of oxidation in the NiCrAlY system. This could include the application of a protective coating, fabrication under inert conditions, preoxidation prior to ceramic deposition, or examination of an alternate structure resistant to oxidation effects.
- 5. Perform limited thermal cycle testing on the zirconia system in a strain isolator configuration.

#### VII. REFERENCES

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TABLE A-1  $\begin{tabular}{ll} \begin{tabular}{ll} \begin{tabula$ 

Brunswick Specimen Description	Density	Strength MPa (KSI)	Modulus GPa (MSI)	Strain
16647-1-2A-1	29.0	26.6 (3.86)	5.58 (.810)	>2.0
16647-1-2A-2	29.6	25.5 (3.70)	7.86 (1.14)	<b>-</b> 3.2
16647-1-2A-3	31.4	18.9 (4.19)	9.24 (1.34)	≥ .82
Average	30.0	27.0 (3.92)	7.56 (1.09)	
16647-2-1A-1	34.99	34.7 (5.03)	8.96 (1.30)	<b>≥1.</b> 0
16647-2-1A-2	36.2	43.1 (6.25)	12.8 (1.86)	≥1.38
16647-1-1A-3	35.06	37.0 (5.36)	13.4 (1.95)	<b>≥1.64</b>
Average	35.4	38.3 (5.55)	11.72 (1.70)	
16647-3-2B-1	39.2	45.4 (6.59)	18.2 (2.64)	<u>≥</u> 1.06
16647-3-2B-2	37.7	45.3 (6.57)	16.3 (2.36)	3.69
16647-3-2B-3	42.9	35.9 (5.20)	10.7 (1.55)	<u>≥</u> 1.63
Average	39.9	42.2 (6.12)	15.1 (2.18)	

TABLE A-2

MEASURED OXIDATION OF BRUNSBOND<sup>R</sup> PAD

TASK II

(1311°K, 24 Hours, Static Air)

							Oxidation	lation	† 
Specimen Description	imen ption	Reference No.	Nominal Surface Area (m <sup>2</sup> )	Wire Surface Area (m <sup>2</sup> )	Pre-weight (kg)	Post-weight (kg)	Nominal Surfaçe (kg/m²)	Weight Percent (%)	Wire Surfaçe (kg/m²)
29.85% Fiber Metal	Metal	Brunswick 16647-1-2A#4	1.75×10 <sup>-3</sup>	1.40x1.0 <sup>-2</sup>	3.5247x10 <sup>-3</sup>	3.5408x10 <sup>-3</sup>	9.14x10 <sup>-3</sup>	+0.45	1.15×10 <sup>-3</sup>
35.85% Fiber Metal	Metal	Brunswick 16647-2-1A#4	1.71×10 <sup>-3</sup>	1.75×10 <sup>-2</sup>	4.4258×10 <sup>-3</sup>	4.45-5x10 <sup>-3</sup>	1.46x10 <sup>-3</sup>	+0.56	1.41x10 <sup>-3</sup>
42.64% Fiber Metal	Meta1	Brunswick 16647-3-28#4	1.70x10 <sup>-3</sup>	2.00x10 <sup>-2</sup>	5.0704x10 <sup>-2</sup>	5.0989x10 <sup>-3</sup>	1.71×10 <sup>-2</sup>	+0.57	1.43x10 <sup>-3</sup>

#### TABLE A-3

#### PLASMA SPRAY PARAMETERS FOR POROUS NICTALY

Parameters	Setting
Spray System Powder Blend (16 hr, 43°K bakeout)	Metco 7M  Task II (-170+325)NiCrA1Y + 10% w/o Metco 600 ≡ 4750 kg/m <sup>3</sup> (-170+325)NiCrA1Y + 25% w/o Metco 600 ≡ 2870 kg/m <sup>3</sup> (-170+325)NiCrA1Y + 30% w/o Metco 600 ≡ 2390 kg/m <sup>3</sup>
	Task III (-100+200)NiCrAlY + 30% w/o Metco 600 $\equiv$ 3010 kg/m <sup>3</sup> Task IV (-100+200)NiCrAlY + 33% w/o Metco 600 $\equiv$ 2347 kg/m <sup>3</sup>
Nozz1e	7MG
Gun Distance	75 mm
Cooling Jets Cross Distance	1.50 mm
Cooling Jets Pressure	20 psi.
Amperage	500
Volts	70
Horizontal Gun Speed	220 mm/sec
Vertical increment	3 mm
1° Arc Gas, Pressure, CFH	N <sub>2</sub> , 50 psi, 100
2° Arc Gas, Pressure, CFH	$H_2^-$ , 50 psi, 5
Powder Feed Rate	20 g/min
Powder Port	2
Deposition Rate	20 μm/cycle
Total Deposition	3.75 mm
Powder Used	2000 g/150 cm <sup>2</sup>

TABLE A-4

MECHANICAL PROPERTY DATA ON PLASMA SPRAYED POROUS NICEALY

TASK II

7	Strain (X)	0.20	0.21	0.35
	Deflectometer GPa (MSI)	23.4 (3.40)	6.21 (0.90)	ı
dus	Deflect	23.4	6.21	1
Wodulus	age (MSI)	52.2 (7.57)	10.5 (1.53)	5.29 (0.77)
	Gage GPa (MSI)	52.2	10.5	5.29
	Strength MPa (KSI)	87.0 (12.6)	(2.83)	(1.91)
4 pt.	Stren	87.0	19.5	13.2
Bulk	Kg/m <sup>3</sup>	4780	2870	2390
	Reference No.	UTRC 80-114	UTRC 80-115	UTRC 80-120

TABLE A-5

OXIDATION DATA ON PLASMA SPRAYED POROUS MICKALY

TASK II

(1311°K for 24 Hours)

on Percent Weight Change (1)	+ 6.1	+ 8.7	7 017
Nominal Percent Weigh Surface (Kg/m <sup>2</sup> ) Change (%)	0.31	0.20	72.0
Post Weight (Kg)	8.5083 x 10 <sup>-3</sup>	$3.6440 \times 10^{-3}$	$4.4013 \times 10^{-3}$
Pre-weight* (Kg)	$8.0228 \times 10^{-3}$	$3.352^4 \times 10^{-3}$	3.9746 x 10 <sup>-3</sup>
Nominal Surface Area (m <sup>2</sup> )	$1.57 \times 10^{-3}$	1.48 x 10 <sup>-3</sup>	$1.56 \times 10^{-3}$
Reference No.	UTRC 80-114	UTRC 80-115	UTRC 80-120

\*The pre-weight represents prior conditioning at  $1000^{
m o}{
m F}$  for 1 hour to remove wax, polyester and moisture.

TABLE A-6

LOW MODULUS CERAMIC 4-POINT BEND PROPERTY TEST RESULTS AT ROOM TEMPERATURE

TASK II

Description	UTRC #	Stre	Strength MPa (KSI)	Gage GPa (MSI)	ge (MSI)	Deflectometer GPa (MSI)	(MSI)	Strain ef (1)	Density p(Kg/m³)
Porous polyester	80-95	1.3	(.19)	3.8	3.8 (.55)	•	ŧ	.05	2270
Microcracked	96-08	8.8	(1.3)	15.1	15.1 (2.2)	9.4	(65.) 0.4	.11	2090
Parameter Variation	80-08	12.7	(1-8)	14.7	14.7 (2.1)	4.5	4.5 (.66)	111.	4930

TABLE A-7

MEASURED OXIDATION OF LOW MODULUS CERAMIC

TASK II

(1311°K, 24 Hours, Static Air)

0xidation	Weight Percent (2)	None	None	None
0xid	Nominal Surfaçe (kg/m <sup>2</sup> )	None	None	None
	Post-weight (kg)	3.7030×10 <sup>-3</sup>	8.3315x10	7.9055x10 <sup>-3</sup>
	Pre-weight* (kg)	3.7090x10 <sup>-3</sup>	8.3327x10 <sup>-3</sup>	7.9060×10 <sup>-3</sup>
	Weight (kg)	4.3293x10 <sup>-3</sup>	8.3462×10	7.9648×10 <sup>-3</sup>
Nominal	Surface Area (m <sup>2</sup> )	1.68×10 <sup>-3</sup>	1.68x10 <sup>-3</sup>	1.68×10
	Reference No.	UTRC 80-95	UTRC 80-96	UTRC 80-98
	Specimen Description	Porous Polyester Zirconia	Microcracked zirconía	Parameter Variation Zirconia

\*For the ceramic systems, the original weight represents the as-machined weight whereas the pre-weight represents prior conditioning at 1000°F for 1 hour to remove moisture and wax (and polyester in the case of specimen 80-95).

TABLE A-8

ROOM TEMPERATURE AND 1039°K MECHANICAL PROPERTY TEST DATA ON 35% BRUNSBOND PAD IN THE AS-RECEIVED CONDITION

TASK III

Temperature	Str	Strength	snInbow	•	Strain to
( <sup>o</sup> K)	MPA	(KS1)	GPA <sup>o</sup>	(MSI)	Failure (Z)
298	37.6	(2.46)	12.1	(1.76)	2.9
	33.0	(4.79)	9.72	(1.41)	2.0
•	36.5	(5.30)	9.66	(1.44)	2.3
Avg.	35.7	(5.18)	10.58	(1.54)	2.4
Temperature (°K)	Str	Strength MPA (KSI)	GPA GPA	GPA (USI) o	Strain to Pasx (Z)
1089°K	2.67	(.416)	4.33	(.628)	13.4
	4.32	(.626)	2.58	(,374)	13.4
	3.53	(.511)	3.96	(.574)	14.3
Avg.	3.57	(*15)	3.62	(.525)	13.7

o Extensometer

TABLE A-9

ROOM TEMPERATURE AND 1089°K MECHANICAL PROPERTY TEST DATA ON 35% BRUNSBOND<sup>R</sup> PAD AFTER THERMAL AGING AT 1311°K FOR 500 HOURS

TASK III

Strain to P max (Z) 4.33 4.2 4.2 3.17	Strain to  max (2)  3.11	4.79
GPA <sup>o</sup> (MSI) <sup>o</sup> 5.82 (.845) 5.15 (.893) 6.9 (2.02) 6.62 (1.25)	Modulus (MSI) <sup>o</sup> (.874) (2.37)	(1.40)
GPA <sup>o</sup> 5.82 6.15 13.9	GPA <sup>o</sup> 6.03	6.53
PA (KSI) .0 (7.10) .4 (5.72) .4 (5.28) .6 (6.03)	ngth (KSI) (.741) (.748)	(.732)
MPA 49.0 39.4 36.4 41.6	MPA 5.11 5.16	5.05
(°K) 298 Avg.	Temperature (°K) 1089	Avg.

o Extensometer

TABLE A-10

ROOM TEMPERATURE AND  $1089^{\circ}$ K MECHANICAL PROPERTY TEST DATA ON POROUS NICLALY (-100 + 200 MESH, UTRC 81-016) DENSITY = 3010 Kg/M³ IN THE AS-SPRAYED CONDITION

TASK III

					SN		
Temperature (°K)	Strength MPA (KSI	ngth (KSI)	Strain Gage GPA	Gage (MSI)	Deflectometer GPA (MS	cometer (MSI)	Strain to to failure 2
298	34.6	(5.02)	15.4	(2.24)	10.0	(1.46)	.35
	23.2	(3.36)	10.2	(1.49)	10.9	(1.58)	.34
	23.5	(3.41)	9.48	(1.38)	9.55	(1.39)	.36
	24.4	(3.54)	11.9	(1.73)	8.52	(1.24)	.31
Avg.	26.4	(3.83)	11.79	(1.71)	9.74	(1.41)	.34
Temperature (°K)	Strength <u>MPA</u> (KSI)	ngth (KSI)			Modu Deflect GPA	Modulus Deflectometer PA (MSI)	
1089	17.6	(2.55)			6.80	(986°)	
	20.1	(2.91)			5.95	(.863)	
	18.0	(2.62)			7.48	(1.08)	
Avg.	18.6	(5.69)	•		6.74	(926.)	

TABLE A-11

ROOM TEMPERATURE AND 1089°K MECHANICAL PROPERTY DATA ON PORGUS NICrAIY (-100 + 200 MESH, UTRC 81-016) DENSITY = 3010 Kg/M<sup>3</sup> AFTER AGING AT 1311°K FOR 500 HOURS

III	
TASK	

	Strain to Failure %	.159	.184	.207	.183						
	Deflectometer (MSI)	(3.62)	(3.03)	(3.38)	(3.38)	Wodulus	Deflectometer A (MSI)	(2.85)	(2.45)	(2.96)	(2.75)
	Defle GPA	25.0	20.9	24.0	23.3	Wod	Deflec GPA	19.7	16.9	20.4	19.0
Wodulus	Strain Gage	(4.37)	(4.44)	(5.47)	(4.76)						
	Si GPA	30.2	30.6	37.7	32.8						*
Strength	(KSI)	(5.63)	(06.9)	(8.50)	(7.01)		(KSI)	(2.40)	(69.9)	(5.20)	(5.76)
SS	MPA	38.8	47.6	58.6	48.3		MPA	37.2	46.1	35.8	39.8
	Temperature (°K)	298			Avg.	Temperature	( <sub>0</sub> K)	1089			Avg.

TABLE A-12

ROOM TEMPERATURE AND 1089°K MECHANICAL PROPERTIES OF AS-SPRAYED MICROCRACKED ZIRCONIA (80-129)

TASK III

	Failure &	.088	.128	.129	.115				
	(NSI)+	(1.21)	(07.)	(89.	(98.)	(.431)	(.541)	(*524)	(667*)
[กส์เน] เกร	GPA* (MSI)* GPA+	8.35	4.85	4.68	5.96	2.97	3.73	3.62	3.44
<u> </u>	(MSI)*	(4.56)	(968.)	(2.23)	(2.56)				
	GPA*	31.4	6.18	15.4	17.7				
rength	(KSI)	(1.61)	(1.29)	(1,32)	(1.41)	(1.64)	(1.79)	(1.82)	(1.73)
St.	MPA	11.1	8.91	9.08	69.6	11.3	12.3	12.5	12.0
	Temperature (°K)	298			Avg.	1089			Avg.

\* Strain Gage

<sup>+</sup> Deflectometer

TABLE A-13

ON MICROCRACKED ZIRCONIA (80-129) AFIER THERMAL AGING AT 1311°K FOR 500 HOURS ROOM TEMPERATURE AND 1089°K MECHANICAL PROPERTY TEST DATA TASK III

Temperature	Stre	angth			5		Create to
(%K)	MPA	MPA (KSI)	GPA*	#(15M)	GPA+	(MSI) <sup>+</sup>	Failure (7)
298	10.7	(1.55)	38.5	(62.5)	19.1	(2.71)	.07
	12.3	(1.78)	26.8	(3.88)	15.8	(2.30)	60*
	10.4	(1.52)	29.3	(4.24)	16.1	(2.33)	.07
Avg.	11.13	(1.61)	31.5	(4.57)	17.0	(5.46)	80.
Temperature (°K)							
1089	10.5	(1.53)			8.52	(1.24)	
	16.4	(2.83)			12.8	(1.85)	
	12.6	(1.82)			9.72	(1.41)	
Avg.	13.2	(2.06)			10.3	(1.50)	

\* Strain Gage

<sup>+</sup> Deflectometer

TABLE A-14

WEIGHT AND DIMENSION CHANGES OF 35% DENSE BRUNSBOND<sup>R</sup> PAD VERSUS TIME AT 1311°K

TASK III

#### Dimensional Change (%)

Time (Hrs.)	X-Y plane*	2-Ax1s**	Weight Change 7
1	+.60	+1.9	+ .34
10	+.20	0	+ .40
100	+.53	+1.9	+ .81
200	20	+3.8	+ .80
500	+.20	-1.0	+1.30

<sup>\*</sup>  $\pm$  1 mil change =  $\pm$  .2%

<sup>\*\*</sup> Z - Axis is thickness direction (100 mils),  $\pm$  1 mil change =  $\pm$  1%.

#### TABLE A-15

## WEIGHT AND DIMENSION CHANGES OF 35% BRUNSPONDR PAD VEPSUS TIME AS THE RESULT OF THERMAL CYCLE TESTING BETWEEN RT AND 1311°K

#### TASK III

Time @ 1311°K(Hr)	Dimensional Ch X-Y Plane	ange (%) Z Plane	Weight Change
1	< ,1.	< 1	+ .18
10	< .1	< 1	+ .36
50	< .1	< 1	+ .54
100	, 2	1	+ .65

TABLE A-16

## WEIGHT AND DIMENSION CHANGES OF POROUS NICRALY (-100 + 200 MESH, UTRC 81-016) DENSITY = 3010 Kg/M<sup>3</sup> VERSUS TIME AT 1311°K

TASK III

#### Dimensional Change (%)\*

Time (Hrs.)	X-Y plane**	2 - Axis***	Weight Change %
1	0	-2.6	+ 1.8
10	0	0	+ 3.1
100	+.06	0	+ 6.9
200	+.33	-1.0	+ 8.2
500	+1.4	+1.8	+10.5

<sup>\*</sup> X-Y plane is perpendicular to Spray Axis, 2 - Axis is parallel

<sup>\*\*</sup>  $\pm$  1 mil change =  $\pm$  .2%

<sup>\*\*\* + 1</sup> mil change + 1%

TABLE A-17

# WEIGHT AND DIMENSIONAL CHANGE MEASUREMENTS OF POROUS N1CrA1Y (-100 + 200 MESH, UTRC 81-016) DENSITY = 3010 Kg/M<sup>3</sup> VERSUS TIME AS THE RESULT OF THERMAL CYCLE TESTING BETWEEN RT AND 131.1°K

#### TASK III

	Dimension	Weight Change	
Time @ 1311°K (hr)	X-Y plane**	Z - Axis***	
1	0	0	+2.8
10	+.59	+1.9	+6.0
50	+.66	+1.9	+7.3
100	+.99	+1.9	+8.3

<sup>\*</sup>X-Y plane = plane perpendicular to spray axis, Z axis = direction parallel to spray axis

<sup>\*\*± 1</sup> mil change = ± .2% \*\*\* ± 1 mil change in thickness = ± 1%

TABLE A-18

WEIGHT AND DIMENSION CHANGES OF MICROCRACKED ZIRCONIA (80-129) VERSUS TIME AT 1311°K

### TASK III

Time (Hrs)	% Change Length (in.)	% Change Width (in.)	% Change Depth (in.)	% Change Weight (g)
0	(1.00)	(.998)	(.100)	(8.47714)
1	0	0	0	0
10	0	0	0	0
106	0	0	0	0
268	0	0	0	0
500	0	0	0 .	0

TABLE A-19

### WEIGHT AND DIMENSIONAL CHANGE MEASUREMENTS OF MICROCRACKED ZIRCONIA (80-129) VERSUS TIME AS THE RESULT OF THERMAL CYCLE TESTING BETWEEN RT AND 1311°K

#### TASK III

Time @ 1311°K (Hr)	Dimensional X-Y Plane	Change (%) * Z Plane	Weight Change
1 .	< .1	< 1	+ .005
10	< .1	< 1	005
50	< .1	< 1	003
100	.3	< 1	-0.0076

<sup>\*</sup>X-Y Plane = plane perpendicular to spray axis

Z Plane = plane parallel to spray axis

TABLE A-20

SPECIFIC HEAT OF TASK III CANDIDATES
(From TPRL 249, Taylor & Groot)

Temp.	Temp.	Temp.	Zirconia (Ws gm <sup>-1</sup> K <sup>-1</sup> )	NiCrAlY (Ws gm <sup>-1</sup> K <sup>-1</sup> )	Fiber Metal (Ws gm <sup>-1</sup> K <sup>-1</sup> )
340	67	152	0.4836	0.4761	0.5011
350	77	170	0.4890	0.4769	0.5081
375	102	215	0.5031	0.4818	0.5206
400	127	260	0.5143	0.4843	0.5314
425	152	305	0.5252	0.4880	0.5426
450	177	350	0.5363	0.4914	0.5537
475	202	395	0.5445	0.4959	0.5650
500	227	440	0.5527	0.5000	0.5740
525	252	485	0.5607	0.5062	0.5891
550	277	530	0.5668	0.5107	0.6005
575	302	575	0.5724	0.5169	0.6128
600	327	620	0.5768	0.5230	0.6245
625	352	665	0.5818	0.5299	0.6357
650	377	710	C.5849	0.5384	0.6454
675	402	755	0.5917	0.5459	0.6584
700	427	800	0.5969	0.5531	0.6624
725	452	845	0.6026	0.5612	0.6909
<b>750</b>	477	890	0.6083	0.5731	0.7244
775	502	935	0.6137	0.5831	0.7661
800	527	980	0.6153	0.5925	0.8243
825	552	1025	0.6200	0.6030	0.8172
850	577	1070	0.6251	0.6141	0.7812
875	602	1115	0.6275	0.6261	0.7472
900	627	1.160	0.6285	0.6331	0.7306
925	652	1205	0.6317	0.6421	0.7162
950	677	1250	0.6345	0.6521	0.6988
975	702	1295	0.6357	0.6632	0.6806

TABLE A-21

THERMAL DIFFUSIVITY OF TASK III CANDIDATES
(From TPRL 249, Taylor & Groot)

Sample	Temp.	Diffusivity
Name	<u>(°C)</u>	$(\operatorname{cm}^2 \operatorname{sec}^{-1})$
	00	0.00/05
Zirconia	23	0.00435
	80	0.00390
	199	0.00352
	322	0.00339
	463	0.00326
	611	0.00320
	730	0.00313
	842	0.00294
NiCrAlY	23	0.00594
	85	0.00533
	200	0.00610
	334	0.00728
	465	0.00782
	598	0.00851
	742	0.00951
	75 <b>1</b>	0.00906
	848	0.00917
Fiber Metal	23	0.00780
	127	0.00789
	254	0.00807
	376	0.00884
	497	0.00876
	626	0.01030
	722	0.01132
	815	0.01302

TABLE A-22

THERMAL CONDUCTIVITY OF TASK III CANDIDATES (From TPRL 249, Taylor & Groot)

Temp. (°C) 23	. Density (gm cm <sup>-3</sup> ) 5.2180 5.2180	Specific Heat (Ws $\overline{gm}^{-1}$ K <sup>-1</sup> ) 0.462	Diffusivity (cm <sup>2</sup> sec <sup>-1</sup> ) 0.00435	Conductivity (W cm <sup>-1</sup> K <sup>-1</sup> ) 0.01049	Conductivity Temp (BIU in hr <sup>-1</sup> ft <sup>-2</sup> F <sup>-1</sup> )(°F) 73 73 6.95	Temp. )(°F) 73
5.2180 5.2180		0.544	0.00352	0.00999	6.93 7.02	392
5.2180		0.592	0.00330	0.01019	7.07	932
5.2180 5.2180		0.624 0.636	0.00316	0.01029	7.13 7.16	1112
5.2180 5.2180		0.645	0,00304	0.01023 0.01016	7.09 7.05	1472 1550
3.0263		0.464	0,00525	0.00737	5.11	73
3.0263		0.496	0,00636	0.00955	6.62	392
3.0263		0.546	0,00750	0.01239	8 .59 8 .80	752
3.0263		0.626	0.00846	0.01603	11.11	1112
3,0263 3,0263 3,0263		0.689 0.697	0.00891 0.00930 0.00944	0.01/88 0.01939 0.01991	13.45 13.81	1472 1472 1550
2.684		0.475	0.00780	0.00994	6,89	73
2.684 2.68%		0.521 0.565	0.00785 0.00800	0.01098 0.01213	7.61 8.41	212 392
2.684		0.613	0.00826	0.01359	9.42	572
2.684 2.684		0.658 0.766	0.00858 0.00900	0.01515 0.01850	10.51	752 932
2.684		0.747	0.01006	0.02017	13.98	1112
2.684		0.681	0.01113	0.02034	14.11	1292
2.684		0.710	•	0.02414	16.74	1472
2.684		0.720	0.01345	0.02599	18.02	1550

TABLE A-23

### AS SPRAYED, ROOM TEMPERATURE AND 1089°K MECHANICAL PROPERTY MEASUREMENTS ON POROUS Nicraly (UTRC 81-043) DENSITY 2347 Kg/M<sup>3</sup>

### TASK IV

Temp.	Str	ength	Mod	lulus	Strain to
State Committee of the	MPa	(KS1)	WPa+ (MSI)+	GPa*(MSI)*	Pmax %
298°K	11.7	(1.69)	3.80 (.55)	7.02 (1.02)	.163
	10.5	(1.52)	2.91 (.42)	4.08 ( .59)	.527
	11.5	(1.66)	2.17 (.31)	5.95 ( .86)	<u>.191</u> .294
Ave.	11.2	(1.62)	2.96 (.43)	5.68 ( .825)	.294
1089°K	9.14	(1.33)	2.71 (.393)		-
	7.89	(1.14)	1.43 (.208)	***** <b>***</b>	-
	7.11	(1.03)	1.17 (.170)	-	<b></b>
Ave.	8.05	(1.17)	1.77 (.257)		

<sup>+</sup>Deflectometer \* Strain Gage

TABLE A-24

ROOM TEMPERATURE AND 1089°K MECHANICAL PROPERTY MEASUREMENTS
ON POROUS NICTALY (UTRC 81-043) DENSITY 2347 Kg/M<sup>3</sup>
AFTER AGING 500 HRS. AT 1311°K

TASK IV

Temp.	Stre	ngth	Modu	lus	Strain to Pmax
	MPa	(KSI)	GPa+(MSI)+	GPa (MSI)*	
298°K	25.6	(3.72)	10.5 (1.53)	19.3 (2.80)	.179
	17.7 18.0	(2.57) (2.61)	8.9 (1.29) 15.9 (2.30)	13.5 (1.96) 20.1 (2.91)	.162 .106
Ave.	20.4	(2.97)	11.8 (1.71)	17.6 (2.56)	.149
1089°K	13.0 11.4	(1.89) (1.66)	10.0 (1.45) 13.0 (1.83)		
Ave	12.2	(1.78)	11.5 (1.67)		

+Deflectometer

\*Strain gage

TABLE A-25

# STATIC OXIDATION OF POROUS NICTALY (DENSITY = 2347 Kg/M³) at 1311°K, WEIGHT AND DIMENSION CHANGES VERSUS TIME

TASK IV

Time at	Wataht Changa	(At the control of th	Dimension Change	The transport of the second second
1311°K (hrs.)	Weight Change	Height	<u>Length</u>	Width
1.	+ 1.70	0	+ .19	0
10	+ 3.87	0	+ .39	+ .53
100	+ 9.33	0	<b>19</b>	+1.55
200	+11.01	0	+1.17	+1.55
500	+16.2	+4.39	+2.58	+2.51

TABLE A-26

## CYCLIC OXIDATION OF POROUS NICTALY (DENSITY = 2347 kg/M<sup>3</sup>) BETWEEN ROOM TEMPERATURE AND 1311°K

TASK IV

		Dimension Change		
Time at 1311°K (hrs.)	Weight Change	Hef. 1	% Length**	Width**
1	+ 2.47	95	59	53
10	+ 3.03	0	.19	0
50	+ 7.58	-1.88	+ .39	+ .52
100	+ 7.99	+1.75	+2.50	+1.28

<sup>\*</sup>  $\pm 1 \text{ mil} = \pm 1\%$ 

<sup>\*\*</sup>  $\pm 1 \text{ mil} = \sim \pm .3\%$ 

TABLE A-27

SPECIFIC HEAT OF NiGraly
(From TPRL249A, Taylor & Groot)

Temp.	Temp.	Temp.	3010 Kg/M <sup>3</sup> Task III NiGrAly	2347 Kg/M <sup>3</sup> Task IV NiCrAlY
(°K)_	(°C)	(°F)	$(Ws gm^{-1}K^{-1})$	$(Ws gm^{-1}K^{-1})$
315	42	108	imo 4,λ	<b>U.4737</b>
320	47	117	,500 BHG	0.4753
325	52	126	-	0.4766
340	67	152	0.4761	prog. soin
350	77	170	0.4769	0.4757
375	102	215	0.4818	0.4864
400	127	260	0.4843	0.4908
425	152	305	0.4880	6.4900
450	177	<b>3</b> 50	0.4914	0.4928
475	202	395	0.4959	0.4932
500	227	440	0.5000	0.4948
525	252	485	0.5062	0.4973
550	277	530	0.5107	0.5003
575	302	575	0.5169	0.5080
600	327	620	0.5230	0.5193
625	352	665	0.5299	0.5246
650	377	710	0.5384	0.5360
675	402	7.55	0.5459	0.5416
700	427	800	0.5531	0.5512
725	452	845	0.5612	0.5598
750	477	890	0.5731	0.5711
775	502	935	0.5831	0.5812
800	527	980	0.5925	0.5903
825	552	1025	0.6030	0.5984
850	577	1070	0.6141	0.6110
875	602	1115	0.6261	0.6212
900	627	1160	0.6331	0.6313
925	652	1205	0.6421	0.6414
950	677	1250	0.6521	0.6498
975	702	1295	0.6632	0.6610

TABLE A-28

THERMAL DIFFUSIVITY OF Nicraly
(From TPRL249A, Taylor & Groot)

Sample	Temp.	Diffusivity (cm <sup>2</sup> sec <sup>-1</sup> )
Nicraly	23	0.00594
3010 кg/м <sup>3</sup>	85	0.00533
Task III	200	0.00610
	334	0.00728
	465	0.00782
	598	0.00851
	742	0.00951
	751	0.00906
	848	0.00917
NiCrAlY	22	0.00435
2347 Kg/M <sup>3</sup>	300	0.00541
Task IV	65.∤	<b>0.00690</b> .
	838	0.00740

TABLE A-29

THERMAL CONDUCTIVITY OF NICLALY